Audit of Data Quality
October 2010 Sampling Event
Data associated with "Ground-Water Investigation in Pavillion, Wyoming," QA ID #G-14478
analyzed at US EPA Region VIII Laboratory
ADQ performed by Neptune and Company, Inc.

ADQ Report Date: August 8, 2011.
Four validation Excel spreadsheets are included in this ADQ report and are provided as separate files: October 2010 Pavillion R8 Volatiles Method 8260 Validation Worksheets, October 2010 Pavillion R8 Semivolatiles Method 8270 Validation Worksheets, October 2010 Pavillion R8 TPH DRO Method 8015D Validation Worksheets, and October 2010 Pavillion R8 TPH GRO Method 8015D Validation Worksheets. These worksheets include documentation of the validation process, along with sample and batch information, and recalculations.

1. **Laboratory Data Audited:**

Laboratory (Organization):
US EPA Region VIII Laboratory.

Sample Type(s)/Methods/Analyte(s): Four analytical methods were to be included in this task for the four samples identified below: 1) TPH/DRO, 2) 8270 semivolatiles, 3) 8260 volatiles and 4) TPH/GRO.

Sample Identification: EPAMW01, EPAMW02, LD01, and RD01.

QA Reviewers: Rebecca Shircliff and David Gratson, Neptune and Company, Inc.

Method Information (all four methods provided as separate pdf files):
1) TPH/DRO: EPA Method 8015D (modified), Region VIII Operating Procedure (OP) ORGM-508 r1.0
2) 8270 semivolatiles: EPA Method 8270D (modified), Region VIII OP ORGM-515 r1.1
3) 8260 volatiles: EPA Method 8260, Region VIII OP ORGM-501 Rev 1.1.

4) TPH/GRO: EPA Method 8015D (modified) Purge and Trap, Region VIII OP ORGM-506 r1.0.

File Information: Final Report included in file 1010008,1010009,1010010 final 02 dec 10.pdf.

TPH/DRO: Associated files: Pavillion #2 LSR 1010-017.pdf and Sequence No. 0J12001.pdf

Semivolatiles via EPA Method 8270: Associated File: Pavillion #2 LSR 1010-017.pdf, Sequence No. 0J25006.pdf and Sequence No. 0J27001.pdf

Volatiles via EPA Method 8260: Associated File: Pavillion #2 LSR 1010-017.pdf and Sequence No. 0J18003.pdf

TPH/GRO: Associated Files: Pavillion #2 LSR 1010-017.pdf, Sequence No. 0J06001.pdf and Sequence No. 0J07001.pdf

QA/QC Criteria for Analytical Methods: QAPP specified and Laboratory specific QA/QC criteria and limits were used as the basis of this ADQ. Note however, the Pavillion QAPP did not provide specific QA/QC criteria associated with the EPA Region VIII Laboratory methods. This QAPP indicates the organic analyses conducted by EPA Region 8 Laboratory are not included other than by reference to the analysis. As such, only corrective actions specified in the EPA Region 8 OPs can evaluated. The laboratory did provide a QA/QC Summary table (attached as a pdf file entitled R8 Lab Summary QA_QC.pdf). The DoD LCS study refers to a study used to derive statistical control limits for Semivolatile and Volatile analytes in laboratory control samples (spiked blank matrix). The QA/QC Summary table, DoD statistical limits, and information gathered during the TSA at Region VIII (unrelated to this project) were used to evaluate the laboratory against data quality indicators and to assess the usability during this ADQ. Table 1 below is a summary of these QA/QC criteria.

Table 1. Region VIII Laboratory QA/QC Requirements.

QC Type	Semivolatiles (Method 8270D)	DRO (Method 8015D)	GRO (Method 8015D)	Volatiles (Method 8260C)	Frequency
Method Blanks	Preparation Blanks (same as method blank), one with each set of extraction groups within the lab, calibration blanks, <RL	Preparation Blanks (same as method blank), <RL	Instrument Blank (IBL) is the method blank <RL	Method Blank <RL	One per sample set

Surrogate Spikes	"System Monitoring Compounds" use DoD derived limits. concentration 5 µg/mL (20 for tribromophenol) with no dilution. Note, for the Pavillion specific compounds, the surrogate 2-fluorophenol limit is 60-120% in the associated laboratory reports.	60-140% of expected value, o-terphenyl	70-130% of expected value, bromofluorobenzene, added automatically by autosampler	Statistical Limits from DoD LCS Study	Every field and QC sample
Internal Standards Verification.	Every sample, EICP area within ±50% of last ICV or first CCV. Add additional IS if needed for dilutions. (SOP Sections 9.4 and 11.4.6)	NA	NA	EICP area within -50% to +100% of ICAL midpoint standard	Every field and QC sample for applicable methods
Initial multilevel calibration	ICAL: minimum of 6 levels (.25 -12.5 µg/L) , one is at the MRL (0.50 µg/L), prior to sample analysis (not daily) RSD≤20%, r^2 ≥0.990	ICAL: 10-500 µg/L RSD<=20% or r^2>=0.990	ICAL: .25-12.5 µg/L for gasoline (different range for other compounds) RSD<=20% pr r^2>=0.990	ICAL, RSD<=20% pr r^2>=0.990	As required (not daily if pass ICV)
Initial and Continuing Calibration Checks	CCV (same source as ICAL): daily and every 12 hours, 80-120% of	Daily with each sequence. ICV1 =DRO, ICV2 = surrogate only check	Daily with each sequence. ICV1 & CCV1 = gasoline, ICV2 & CCV2 = BTEX+MTBE+naphthalene	ICV (second source) % R ±20% CCV % R ±20%	CCV At beginning of sample set, every tenth sample, and end of sample set

	expected value	80-120% of expected value	80-120% of expected value		
Second Source Standards	ICV1 is from a second source (includes 7 special compounds) Once after each ICAL, 70-130% of expected value	ICV1 is from a second source, 80-120% of expected value	ICVs are from different source. 80-120% of expected value	ICV (second source) % R ±20%	Each time calibration performed
Standard Reference Material (SRM)	Once per batch, limits based on SRM certification	See below	See below	NA	
Laboratory Control Samples (LCS)	Blank Spike, one per extraction group included once per sequence or every 20 samples. 1mL into 1 L of sample at mid level. Statistical Limits from DoD LCS Study (rounded to 0 or 5)	Often use SRM as LCS, if so limits based on certification information, otherwise 70-130% of expected value	Often use SRM as LCS, if so limits based on certification information, otherwise 70-130% of expected value.	Spike Recovery within Statistical Limits from DoD LCS Study	One per analytical batch or every 20 samples, whichever is greater
Matrix Spikes (MS)	Same as LCS	Same as LCS (70-130%, may develop statistical based in future)	Spike with ICAL mix Gasoline 70-130%, others DoD limits	Spike Recovery within Statistical Limits from DoD LCS Study	One per sample set or every 20 samples, whichever is more frequent
MS/MSD	Once per batch or every 20 samples. RPD ≤ 20% Note, the limits in the Reg VIII lab files is ≤ 30%	RPD ≤ 25	RPD ≤ 25	RPD≤30%	One per sample set or every 20 samples, whichever is more frequent
Detection Limit Standard (CRL)	run MDL study approximately annually	DL= RL, ICAL run down to 10 µg/L MDL study	DL= RL, MDL study approximately annually	±50% of expected value	CRLs not routinely analyzed, only report to RL (lowest standard of cal

		approximately annually			model)
Reporting Limits*	0.1 µg/L (generally)[1]	20 µg/L[1]	Gasoline is 20 µg/L[2] Other compounds RL is from 1-200, compound specific	Not specified in QAPP, as EPA RSK was doing the analysis for Killdeer	NA (part of ICAL)
Other Method Specific	GC/MS tuning (DFTPP) : prior to ICAL and at beginning of each 12-hour period.			GC/MS tuning (BFB): prior to ICAL and at beginning of each 12-hour period.	

[1]Based on 1000 mL sample to 1 mL extract

[2]Based on a 5 mL purge

*these limits are compound dependent (see table below)

2. Summary of Assessment

Note, the terms Findings and Observations are based upon the definitions in the SOP LSAS-QA-02-0, Performing Audits of Data Quality.

OBSERVATIONS

1. **Recalculations Do Not Match Reported Values.** The recalculated values for several 8260 VOC and 8270 SVOC compounds do not match the reported values. In some cases this is likely due to the laboratory using a quadratic or linear equation that was not provided. However, those analytes that indicate the calibration was based upon an average response factor should match our recalculated values. See Table below and individual spreadsheets. The largest differences, where the average RF is used, are noted for phenol (reported at 10.7 µg/L, recalculated at 12.3 µg/L), ethylbenzene (reported at 67 µg/L, recalculated at 72 µg/L), o-xylene (reported at 178 µg/L, recalculated at 204 µg/L).

2. **Corrective Actions for QC.** The corrective actions the laboratory takes when QA/QC is not met is not always specified, none are specified in the analytical methods.

3. **SVOC.**
   a. Samples between CCV1 and CCV2 should be qualified due to low (60%) recovery of the analyte 3,3'dichlorobenzidine in CCV1.
   b. The surrogate 2-fluorobiphenyl recovery of 48.8% is just below the recovery limits (50%) in the EQ Blank. The standard approach is to qualify all compounds (not just detects) in the EQ Blank associated with this surrogate. All compounds are qualified because the recovery was below the limit, indicating the potential for a false negative. This approach is also consistent with the Region VIII flagging criterion.

4. **GRO.** The raw data for batch 1000476 for the GRO analysis was not located in the laboratory reports.

EDITORIAL COMMENTS

1. **DRO Analysis Method.** The results report for DROs lists 8015**B** as the analysis method, see Question 7 below in table. This should be 8015D.

ADDITIONAL QUALIFIERS BASED ON FIELD QC

1. **SVOC (all units µg/L):**
   a. Benzyl alcohol was found in the field, trip blank, and EQ blank in the range of .63-to approximately 1 µg/L. Samples with detects should be qualified. Carefully evaluate those samples with benzyl alcohol in this same concentration range.
   b. Benzoic acid was found at <1 µg/L in the EQ Blank. Samples with detects should be qualified. Carefully evaluate those samples with benzoic acid in this same concentration range.

c. Squalene was found in the run, prep, trip, and EQ up to 0.49. Samples with detects should be qualified. Carefully evaluate those samples with squalene in this same concentration range.
d. 2-butoxy ethanol phosphate was found in the EQ Blank at 2.53 µg/L. Samples with detects should be qualified. Carefully evaluate those samples with 2-butoxy ethanol phosphate in this same concentration range.
e. Adamanatane was found in the EQ Blank at 0.32 µg/L. Samples with detects should be qualified. Carefully evaluate those samples with adamanatane in this same concentration range.

ITEMS REVIEWED

Number	ADQ Issue	Yes	No	NA	Comments
File Information					
1	Provide File names: See Inventory of Document-N&C.doc file, provided with this report.				
Sample Information					
3	Are samples uniquely identified and correctly transcribed throughout the data package to the summary of results?	**X**			Samples are uniquely labeled as EPAMW01, EPAMW02, LD01, and RD01 for all methods. In addition, samples are identified by unique Lab IDs throughout the raw data packages for all methods.
4	Does sample collection documentation indicate that samples were collected as described in the QAPP, and the schedule and volumes in the planning documentation?		**X**		The only sample collection documentation within the report files is: date/time sample was collected, sample volume and pH for DROs and number of samples collected. Any additional specific sampling information is not expected to be in the laboratory report. So, this is acceptable.
5	Does sample collection documentation indicate appropriate preservation?	**X Partial**			According to the Pavillion QAPP, none of the samples for Reg VIII were to be acidified in the field. DRO samples were acidified upon receipt at the lab for analysis. All samples were preserved on ice during shipment. There is no clear indication of how the GRO and DRO samples were preserved after receipt by the labs (e.g. temperature stored at). However, the case narrative for VOCs and SVOCs indicates that they were preserved at 4 $\pm$ 3°C.